# PATENT ABSTRACTS OF JAPAN

(11)Publication number:

2001-026246

(43) Date of publication of application: 30.01.2001

(51)Int.CI.

B60R 21/04 B32B 5/18 C08J 9/00 F16F 7/00 // B29C 44/00 B60R 13/02 B29K 67:00

(21)Application number: 11-

(71)Applicant: SEKISUI PLASTICS

198021

CO LTD

(22)Date of filing:

12.07.1999 (72)Inventor: EGUCHI TATSUYA

EGUCHI TATSUTA

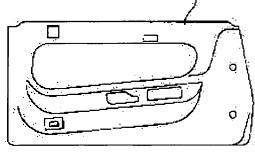
**ONO YASUHIRO** 

# (54) INTERIOR MATERIAL FOR AUTOMOBILE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an interior material for automobile with a constant impact absorbing performance by providing a core material formed of the foam of a synthetic resin.

SOLUTION: This interior material for automobile comprises a core material 1 formed of the foam of a synthetic resin, and a ratio (S2/S1) of a minimum value S1 and a maximum value S2 of 5% compression strength in a range of an environmental temperature of -20 through +50° C, of the core material is below 3. A thermoplastic polyester resin is used for the foam molding



used in the core material. By using the thermoplastic polyester resin with a reduced speed of crystallization, a degree of crystallization of pre-

expanded particles can be prevented from being increased in excess, the forming fusion in the internal die foam molding can be prevented from being extremely lowered, and the foam molding can be easily manufactured under ordinary molding conditions by using a general foam molding machine.

## **LEGAL STATUS**

[Date of request for examination]

10.05.2002

[Date of sending the examiner's

07.12.2004

decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

## \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

# **CLAIMS**

# [Claim(s)]

[Claim 1] Interior material for automobiles characterized by consisting of foam of synthetic resin and having the core material whose ratios S2/S1 of the minimum value S1 of 5% compressive strength and maximum S2 in a temperature requirement with an environmental temperature of -20-+50 degrees C are three or less.

[Claim 2] Interior material for automobiles according to claim 1 whose degree of crystallinity of the is 20 – 40% and whose rate of welding the core material is formed with the foaming object which carried out foaming in a mold of the pre-expansion particle of thermoplastic polyester system resin, and is 40% or more.

[Claim 3] Interior material for automobiles according to claim 2 which is what contains at least one sort of components chosen from the group to which thermoplastic polyester system resin consists of isophthalic acid and cyclohexane dimethanol into [all] a component in 0.5 – 10% of the weight of the range in a total amount.

[Claim 4] Interior material for automobiles according to claim 2 or 3 some of whose thermoplastic polyester system resin [ at least ] is playback resin collected from the used product.

# [Translation done.]

## \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

## **DETAILED DESCRIPTION**

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the interior material for automobiles.

[0002]

[Description of the Prior Art] For example, a door inside, a door trim, a head-lining inferior surface of tongue, a rear package, a knee bolster, An air bag door, a headrest, an armrest, various pillars, a quarter trim, A frontside trim, a front seat back, a crush pad, A console box, a console lid, RAGGEJI floor covering, The core material which consists of a foaming object in a mold made of a polypropylene regin recently as interior material for automobiles, such as a partition board, a center console, and a lid of a console box, The thing equipped with the epidermis material of the polyolefine system resin foaming sheet of high expansion ratio which wraps the core material concerned entirely, and the product made from the polypropylene regin sheet by which the laminating was carried out to the outside was proposed (for example, refer to JP,6–115025,A). [0003]

[Problem(s) to be Solved by the Invention] However, the foam of the polypropylene regin which constitutes a core material in the above—mentioned interior material for automobiles according to the place which artificers examined Although excelled in lightweight nature, cracking resistance, adiathermic, chemical resistance, etc., since the temperature dependence of a mechanical strength, especially compressive strength is large For example, the thing for which the temperature dependence of the

engine performance which absorbs the impact is [ that the operator and those / attendance / who have ridden on inside should be taken care of ] high at the time of the collision of an automobile etc., and it runs short of the impact absorptivity ability under hot environments especially, So, it became clear for the need for using the spec. design and the foam with a consistency big concrete more in consideration of the fall on the strength by the temperature rise etc. to arise, and to produce the various problems that sufficient lightweight-ization cannot be attained.

[0004] The purpose of this invention is to offer the interior material for automobiles which has related almost fixed impact absorptivity ability to temperature.

# [0005]

of a collision etc.

[Means for Solving the Problem] In order to solve the above-mentioned technical problem, artificers examined whether the interior material for automobiles which has related almost fixed impact absorptivity ability to temperature would be obtained, when the core material of the interior material for automobiles had what kind of property. about [ consequently, / which interior material experiences when an automobile is usually boiled and is used through every year / which is predicted to come out ] — with [ the ratios S2/S1 of the minimum value S1 of 5% compressive strength and maximum S2 in an about -20-+50-degree C temperature requirement ] three [ or less ] It came to complete a header and this invention for the ability of the interior material for automobiles using such a core material to maintain related almost uniformly to temperature the engine performance which absorbs an impact at the time

[0006] That is, the interior material for automobiles of this invention consists of foam of synthetic resin, and is characterized by having the core material whose ratios S2/S1 of the minimum value S1 of 5% compressive strength and maximum S2 in a temperature requirement with an environmental temperature of -20-+50 degrees C are three or less. [0007]

[Embodiment of the Invention] Below, this invention is explained. <Interior material for automobiles> The interior material for automobiles of this invention is equipped with the core material which consists of foam of synthetic resin as mentioned above, and it is characterized by the ratios S2/S1 of the minimum value S1 of 5% compressive strength and maximum

S2 in the temperature requirement with an environmental temperature of -20-+50 degrees C of the core material concerned being three or less. [0008] That to which the above-mentioned ratios S2/S1 exceed 3 produces the problem of changing sharply the impact absorptivity ability at the time of a collision with environmental temperature, as mentioned above. In addition, in order to stabilize further the impact absorptivity ability of the interior material for automobiles to change of environmental temperature, it is desirable that the above-mentioned ratios S2/S1 are two or less, and it is still more desirable that it is 1.5 or less. [0009] In addition, the lower limit of ratios S2/S1 cannot be overemphasized, and is 1 when the above-mentioned minimum value S1 and maximum S2 are equal. Although many things are considered as foam of synthetic resin which satisfies this temperature-compressive strength property, the foaming object which carried out foaming in a mold of the pre-expansion particle of thermoplastic polyester system resin especially is used suitably.

[0010] When satisfying the above-mentioned temperature-compressive strength property, the foaming object of thermoplastic polyester system resin is lightweight, and excellent in impact absorptivity, a repeat compression set, chemical resistance, etc. moreover, especially the foaming object of the above-mentioned thermoplastic polyester system resin be excellent also in the dimensional stability under hot environments, and have the advantage that the interior material for automobiles which cannot generate easily deformation, a camber, etc. by gap of a dimension with other members, such as a sheet of the epidermis material describe below and a late-coming bubble scale factor and a configuration maintenance material, and a configuration etc. and which can fully be equal to long-term use can be constitute.

[0011] The interior material for automobiles covers with epidermis material as usual the part of the above-mentioned core material exposed to the interior of a room of an automobile at least, and is formed. As epidermis material, flexible web materials, such as a polypropylene regin sheet mentioned above, are used suitably. Moreover, there is an advantage that the feel when pressing interior material etc. can be improved, maintaining the impact absorptivity ability by the core material as it is, if the polyolefine system resin foaming sheet of high expansion ratio mentioned above is infixed between epidermis material and a core material.

[0012] Moreover, in order to maintain the configuration of the whole, you may also include the configuration maintenance material formed tabular, in the shape of a frame, etc. in the interior material for automobiles. In addition, various design changes can be performed in the range which does not change the summary of this invention.

<Thermoplastic polyester system resin> Each conventionally well-known various thermoplastic polyester system resin represented by the polyethylene terephthalate (PET) compounded by carrying out the polycondensation reaction of a terephthalic acid and the ethylene glycol, for example as the aforementioned thermoplastic polyester system resin which forms the foaming object used suitable for a core material in this invention is usable.

[0013] However, in order to require time amount high [ conventional thermoplastic polyester system resin, such as Above PET, / generally ] gas barrier nature and great although a foaming agent is sunk in a foaming agent is sunk into resin — making — [a sinking—in process], while heating subsequently and carrying out pre—expansion After particle—izing and obtaining a pre—expansion particle, it is filled up with a [pre—expansion process] and this pre—expansion particle in a mold. There is a possibility of taking time amount, cost, and time and effort with a foaming object, i.e., having manufactured the core material of the interior material for automobiles in this case, by the conventional foaming approach which is made carrying out heating expansion and carries out foaming [the foaming process in a mold].

[0014] Crystallization tends to progress with heating, i.e., since the rate of crystallization is quick, the degree of crystallinity of a pre-expansion particle becomes high too much with heating at the time of abovementioned sinking in and pre-expansion, at the time of foaming in a mold, the fall of the foaming welding nature of foaming grains is imitated, and the thermoplastic polyester system resin of further the above-mentioned former also has a problem of \*\*. For this reason, in having used the general-purpose foaming machine, for example, the gage pressure of steam having carried out foaming by the usual process condition of 0.5 or less MPas especially, although the foaming object excellent in thermal resistance is acquired, foaming grains cannot manufacture the foaming object which was excellent in reinforcement, such as welding and said unified compressive strength, good in it being also at the high rate of

welding.

[0015] Therefore, in order to manufacture the foaming object which has a high rate of welding using conventional thermoplastic polyester system resin, such as PET, the need of fabricating by special process condition to which gage pressure exceeds 0.5MPa(s) using the special foaming machine which gave the special function of being able to supply a lot of steam in a mold at homogeneity is produced. However, such a special process condition, therefore the foaming object manufactured become the thing which has too high degree of crystallinity and which exceeds 40%, although excelled in thermal resistance, it becomes weak, and the reinforcement needed on the contrary is not obtained.

[0016] Moreover, the dimensional stability under hot environments falls and especially the foaming object with which degree of crystallinity exceeded 40% also has the problem of being easy to produce deformation of interior material, camber, etc. So, especially in this invention, it is desirable to use that by which the rate of the crystallization was controlled as the above-mentioned thermoplastic polyester system resin. That is, it is controlled that the degree of crystallinity of the pre-expansion particle according [ the thermoplastic polyester system resin with which the rate of crystallization was controlled ] to heating compared with the conventional PET etc. becomes high too much, and it is prevented that the foaming welding nature at the time of foaming in a mold becomes remarkably low.

[0017] Therefore, without using a special foaming machine for the foaming object excellent also in dimensional stability, while excelling in the foaming welding nature at the time of foaming in a mold and excelling also in a mechanical strength, the usual process condition which used the general–purpose foaming machine enables it to manufacture easily, and it can control that the degree of crystallinity of the acquired foaming object becomes high too much.

[0018] A differential scanning calorimeter (DSC) is used for the rate of crystallization of thermoplastic polyester system resin, and it is Japanese Industrial Standards JIS. The peak temperature (temperature of the peak from which crystallization arises at the time of a temperature up) of crystallization of the resin measured according to the measuring method of K7121 printing can estimate. That is, resin can need that is, say a lot of heat that the rate of crystallization is slow to promoting crystallization, so

that the peak temperature of crystallization is high.

[0019] The above-mentioned crystallization peak temperature is measured filling up the measurement container of DSC with the thermoplastic polyester system resin of the specified quantity as a test portion, and specifically carrying out a temperature up with 10-degree-C programming rate for /. Thus, if the range of the crystallization peak temperature of the measured thermoplastic polyester system resin is about 130 degrees C or more, it can be said that it is suitable thermoplastic polyester system resin with which the rate of crystallization was controlled as mentioned above.

[0020] In addition, as for especially crystallization peak temperature, it is desirable that within the limits of the above is also 180 degrees C or less. When crystallization peak temperature exceeds 180 degrees C, since the glass transition point of resin becomes high, the condition width of face of foaming in a mold becomes narrow, and when shaping becomes less easy on the contrary, there is also a possibility of producing the problem that become easy to generate contraction on the surface of a Plastic solid, and the good foaming object of an appearance is not acquired at the time of foaming in a mold. Moreover, the problem that the manufactured foaming object will become weak may also be produced.

[0021] In addition, when it takes into consideration manufacturing a good pre-expansion particle and a good foaming object in consideration of the balance of each above-mentioned property, as for especially the peak temperature of thermoplastic polyester system resin, it is desirable that within the limits of the above is also about 132–175 degrees C, and it is still more desirable that it is about 135–170 degrees C. As thermoplastic polyester system resin with which are satisfied of this property, although not limited to this, what contains at least one sort of components chosen from isophthalic acid and the group which consists of cyclohexane dimethanol in 0.5 – 10% of the weight of the range in a total amount is raised into [ all ] a component.

[0022] That is, it is formula (1): [0023] as dicarboxylic acid.

[Formula 1] HOOC COOH

[0024] While coming out and using these both together as diol, using

cyclohexane dimethanol, using the isophthalic acid expressed When using either independently and using both together again, a content rate independent [ the ] the content rate of the sum total The above—mentioned thermoplastic polyester system resin made into 0.5-10% of the weight of within the limits in [ all ] a component, respectively Since crystallization peak temperature becomes within the limits which is 130-180 degrees C according to the operation which controls crystallization of resin which isophthalic acid and/or cyclohexane dimethanol have, it becomes possible to manufacture the good foaming object which does not produce the above various problems.

[0025] In addition, when it takes into consideration manufacturing a good pre-expansion particle and a good foaming object in consideration of the balance of each above-mentioned property, as for isophthalic acid and/or the content rate of cyclohexane dimethanol, it is desirable that especially within the limits of the above is also about 0.6 - 9.5 % of the weight, and it is still more desirable that it is about 0.7 - 9 % of the weight. formula (2): [0026 which two methanol parts permuted by the 1st place of a cyclohexane ring, and the 4th place fundamentally as cyclohexane dimethanol among the above, respectively — ]

[0027] Although it comes out and 1 expressed and 4-cyclohexane dimethanol are used, if the isomer permuted by other locations of a cyclohexane ring also has two little methanol parts, it can use together. As dicarboxylic acid, a terephthalic acid, a phthalic acid, etc. are raised, for example among other components which constitute thermoplastic polyester system resin with the above-mentioned isophthalic acid and cyclohexane dimethanol.

[0028] Moreover, as a diol component, ethylene glycol, alpha-butylene glycol (1, 2-butanediol), beta-butylene glycol (1,3-butanediol), tetramethylene glycol (1,4-butanediol), 2, 3-butylene glycol (2, 3-butanediol), neopentyl glycol, etc. are raised, for example.

[0029] moreover, in the raw material of thermoplastic polyester system resin It adds to each above-mentioned component. As an acid component as the multiple-valued carboxylic acid, its anhydride, or the alcoholic components more than trivalence, such as tetracarboxylic acid, such as

tricarboxylic acid, such as trimellitic acid, and pyromellitic acid the range which does not affect the crystallinity of thermoplastic polyester system resin, a rate of crystallization, etc. which mentioned above the polyhydric alcohol more than trivalence, such as tetra-oar, such as triol, such as a glycerol, and pentaerythritol, etc. — small quantity — you may make it contain

[0030] The above-mentioned thermoplastic polyester system resin is manufactured by carrying out the polycondensation reaction of the raw material which predetermined was comparatively got blocked in each above-mentioned component, and contained isophthalic acid and/or cyclohexane dimethanol in 0.5 – 10% of the weight of the range in the total amount as mentioned above as usual. Moreover, the above-mentioned thermoplastic polyester system resin blends two or more sorts of thermoplastic polyester system resin with which isophthalic acid differs from the content rate of cyclohexane dimethanol so that the content rate of the isophthalic acid and/or cyclohexane dimethanol which are occupied in [ all ] a component may become in a total amount 0.5 – 10% of the weight of within the limits, for example, it can manufacture it also melting and by mixing under heating using an extruder etc.

[0031] According to this approach, the content rate of both the above—mentioned components in the manufactured pre—expansion particle can be adjusted only by changing the blending ratio of coal of two or more sorts of thermoplastic polyester system resin with which isophthalic acid differs from the content rate of cyclohexane dimethanol in the manufacture phase of a pre—expansion particle. For this reason, tuning can be simplified compared with the case where the content rate of both components is adjusted in the synthetic phase of resin, and there is an advantage that it can respond now to modification of a specification etc. flexibly.

[0032] Moreover, there is also an advantage of becoming possible to attain effective reuse-izing of a resource, streamlining of dust, and low cost-ization of a pre-expansion particle, by using the ingredient collected and reproduced from the used PET bottle etc. as one sort of the thermoplastic polyester system resin blended, for example. In addition, in the above-mentioned approach, melting and mixing under heating are fully desirable so that each resin may alloy-ize by the ester exchange reaction between two or more sorts of thermoplastic polyester system resin and it may become uniform thermoplastic polyester system resin.

[0033] In addition, after pre-expansion is carried out, in [ which was mixed with the foaming agent under high-pressure melting using the extruder etc.] cutting subsequently and manufacturing, in advance of mixing of a foaming agent, it performs production of melting of two or more sorts of resin, and the uniform thermoplastic polyester system resin by mixing in the above-mentioned extruder as mentioned above, so that a pre-expansion particle may be mentioned later, and continuously, it is efficient to enforce the above-mentioned manufacture approach, and, subsequently it is desirable.

[0034] However, melting and the uniform thermoplastic polyester system resin mixed and produced may be beforehand fed into an extruder for two or more sorts of resin using another equipment, and a pre-expansion particle may be manufactured by the above-mentioned manufacture approach. In addition, when melting at the time of manufacturing a pre-expansion particle, miscibility, the moldability at the time of manufacturing a foaming object by foaming in a mold using the manufactured pre-expansion particle, etc. are taken into consideration, as for the thermoplastic polyester system resin used by this invention, it is desirable that the intrinsic viscosity (measurement temperature: 35 degrees C, solvent:orthochromatic chlorophenol) is 0.6 to about 1.5.

<Pre-expansion particle> As usual, after infiltrating a foaming agent into the above-mentioned thermoplastic polyester system resin, it may particle-ize and a pre-expansion particle may be manufactured while heating and carrying out pre-expansion.

[0035] However, while skipping the process which infiltrates a foaming agent into thermoplastic polyester system resin and saving time amount, cost, and time and effort In order to make still lower the degree of crystallinity of the pre-expansion particle manufactured and to control further the fall of the foaming welding nature at the time of foaming in a mold As mentioned above, after mixing with a foaming agent under high-pressure melting, carrying out pre-expansion of the above-mentioned thermoplastic polyester system resin and acquiring a pre-expansion object, it is desirable to cut this and to manufacture a pre-expansion particle.

[0036] The extrusion-foaming method using the extruder as the approach of mixing with a foaming agent and carrying out pre-expansion of the thermoplastic polyester system resin under high-pressure melting, is

efficient, and is adopted suitably. Although it may be a single screw extruder, a twin screw extruder, etc. which especially the extruder that can be used is not limited but are usually used for extrusion-foaming shaping of this kind and you may be the tandem die which connected these further, the extruder which has sufficient melting and mixed capacity is desirable.

[0037] Things various as a mouthpiece of an extruder can be used for example, a circular ring-like mouthpiece and a flat -- a mouthpiece and a nozzle -- a mouthpiece and the multi-nozzle by which further two or more nozzles have been arranged -- a mouthpiece etc. is raised. These mouthpieces can be used and pre-expansion objects of various configurations, such as the shape of the shape of a sheet, tabular, and a rod, can be made. Various approaches are adopted in order to make a preexpansion object into the predetermined configuration mentioned above. [0038] for example, in order to acquire a sheet-like pre-expansion object, a mandrel top is gone on in the pre-expansion object of the shape of a cylinder extruded from the circular ring-like mouthpiece -- making -considering as the shape of a sheet \*\*\* -- a flat -- what is necessary is just to make into the shape of a sheet a tabular pre-expansion object with the thickness extruded from the mouthpiece with a chilled roll Moreover. in order to acquire a thick tabular pre-expansion object, foaming is advanced making it close to the metal plate of a pair, and the approach of making it into predetermined thickness etc. is adopted. [0039] Various approaches, such as making the cooling system by which the temperature control was carried out besides air cooling or water cooling contact as the cooling approach of a pre-expansion object etc., can be used. It is important to control that perform cooling of a preexpansion object as promptly as possible, and crystallization advances too much. Thus, a pre-expansion particle is completed by cutting the preexpansion object of the manufactured various configurations, and making it into the shape of cylindrical, corniform, and a chip etc. suitably. [0040] Cooling and cutting of the above-mentioned pre-expansion object can be performed to proper timing. For example, after cooling through the pre-expansion object extruded from the mouthpiece underwater at the time of the arbitration under foaming thru/or after the completion of foaming, you may cut in a predetermined configuration and magnitude using a pelletizer etc. Moreover, it is just before the completion of foaming

extruded from the mouthpiece, or immediately after the completion of foaming, and you may cool, after cutting the pre-expansion object before cooling immediately.

[0041] Furthermore, once rolling round the pre-expansion object extruded in the shape of a sheet in the shape of a roll and keeping it by a winder etc., a grinder and a cutting machine may cut it. The magnitude of the pre-expansion particle manufactured in this way is expressed with mean particle diameter, and its about about 0.5-5mm is desirable. Moreover, when a foaming machine general-purpose as mentioned above is used for it, and carries out foaming by the usual process condition, and it takes into consideration acquiring the high foaming object of a mechanical strength excellent in the welding nature of particles, as for the degree of crystallinity of a pre-expansion particle, it is desirable that it is about 8% or less extent.

[0042] moreover, the time of building a pre-expansion particle — yet — remaining heat — \*\*\*\* — in order to prevent becoming easy to fuse the pre-expansion particles which are, as for the above-mentioned degree of crystallinity, it is desirable that it is about 1% or more. In addition, as for especially the degree of crystallinity of a pre-expansion particle, it is desirable that within the limits of the above is also about 1 — 7%, and it is still more desirable that it is about 1 — 6%.

[0043] A differential scanning calorimeter (DSC) is used for degree of crystallinity (%) as well as measurement of the crystallization peak temperature described previously, and it is Japanese Industrial Standards JIS. From the amount of cold heat of crystallization and the amount of heat of fusions which were measured according to the measuring method of K7121 printing, it asks by the degree type.

[0044]

```
      [Equation 1]

      結晶化度(%) =
      モルあたりの融解熱量ーモルあたりの冷結晶化熱量
完全結晶PETのモルあたりの融解熱量
```

[0045] In addition, the amount of heat of fusions of perfect crystal PET per mol in a formula is set to 26.9kJ(s) from the publication of a giant—molecule data handbook [the Baifukan issue]. Specifically the measurement container of DSC is filled up with the pre-expansion particle of the specified quantity as a test portion, the amount of cold heat of crystallization and the amount of heat of fusions are measured, carrying

out a temperature up with 10-degree-C programming rate for /, and the degree of crystallinity of a pre-expansion particle is called for from the measurement result based on the above-mentioned formula. [0046] Although the bulk density of a pre-expansion particle can adjust the pre-expansion particle concerned suitably according to the consistency of the foaming object manufactured by carrying out foaming in a mold etc., it is usually desirable that it is bulk density almost equal to a foaming object. Various additives may be added to a pre-expansion particle. As an additive, for example, a cellular regulator, a flame retarder, an antistatic agent, a coloring agent, etc. are raised other than a foaming agent. in order [ moreover, ] to improve the melting property of thermoplastic polyester system resin — the metallic compounds of Ia like an epoxy compound like glycidyl phthalate, an acid anhydride like pyromellitic acid 2 anhydride, and a sodium carbonate, and an IIa group etc. -- a modifier -- carrying out -- a simple substance -- or two or more sorts can be mixed and it can add. In order that they they not only improve the fizz of a pre-expansion particle, but may improve the rate of a closed cell of the obtained foaming particle, since especially these modifiers can enlarge expansion force of a pre-expansion particle, they are effective.

[0047] These any may be used, although it will be classified into the solidstate compound which decomposes at the temperature more than the softening temperature of thermoplastic polyester system resin, and generates gas, the liquid evaporated within thermoplastic polyester system resin, the inactive gas which may be dissolved in thermoplastic polyester system resin under pressurization as a foaming agent which can be used by this invention if it divides roughly, and it heats. Among these, as a solid-state compound, an AZOJI carvone amide. dinitrosopentamethylenetetramine, a hydra ZORUJI carvone amide, sodium bicarbonate, etc. are raised, for example. moreover -- as the liquid to evaporate -- a propane, n-butane, an isobutane, n-\*\* NTAN, and an isopentane -- it passes and an ether compound like saturated aliphatic hydrocarbon like KISAN, \*\* NZEN, a xylene, aromatic hydrocarbon like toluene, a methyl chloride, halogenated hydrocarbon like Freon (trademark), wood ether, and methyl tert-butyl ether etc. is raised. As a still more nearly inactive gas, a carbon dioxide, nitrogen, etc. are raised, for example.

[0048] In addition, when cutting and manufacturing a thermoplastic polyester system resin pre-expansion particle, after mixing with a foaming agent under high-pressure melting, extruding a pre-expansion particle and carrying out pre-expansion using an extruder, as mentioned above, while evaporating at the moment of extruding from the mouthpiece of an extruder and making melting resin foam, it is desirable to use the foaming agent which takes the heat of the melting resin concerned, for example, saturated aliphatic hydrocarbon, halogenated hydrocarbon, etc. Since these foaming agents are effective in carrying out the operation which cools the fused thermoplastic polyester system resin, and stopping the degree of crystallinity of a pre-expansion particle low, they are desirable. [0049] Moreover, to a pre-expansion particle, it is the range which has big effect neither on the crystallinity of thermoplastic polyester system resin, nor the rate of crystallization, for example, thermoplastic elastomer, such as polyolefine system resin, such as a polypropylene regin, and a polyester system, a polycarbonate, an ionomer, etc. may be added. As an approach of manufacturing the core material of the interior material for automobiles as a foaming object using a pre-expansion particle, the approach of filling up with a pre-expansion particle the metal mold which cannot be sealed although it can be closed down, and introducing and carrying out foaming in a mold of the steam as a heating medium further is desirable. [0050] As a heating medium at this time, although hot blast, oil, etc. can be used besides steam, when fabricating efficiently, steam is the most effective. What is necessary is just to pick out the fabricated foaming object from metal mold, after cooling. What is necessary is to use a general-purpose foaming machine, as mentioned above, and just to carry out foaming by the usual process condition, in carrying out foaming in a mold with steam, after [ namely, ] being filled up with a pre-expansion particle to metal mold -- first -- low voltage -- fixed time amount and steam are carried out with [for example, gage pressure 0.04MPa extent], and external HE discharge of Ayr between particles is carried out by HE \*\*\*\*\*\* in metal mold. Subsequently, the pressure up [for example, 0.08MPa extent] of the \*\* of the steam to blow is carried out, and while making a pre-expansion particle foam in a mold, particles are made to be able to weld and it can consider as a foaming object.

[0051] Moreover, after putting a pre-expansion particle into a well-closed container beforehand and pressing inert gas, such as carbon dioxide gas,

nitrogen, and helium, fit, until just before using it for foaming in a mold in metal mold, by holding under the ambient atmosphere of the gas pressed fit, expansion force at the time of foaming in a mold in the metal mold of a pre-expansion particle can be enlarged more, and a good foaming object can also be acquired. As for especially the rate of welding used as the criteria of the welding nature of particles in the foaming object acquired in this way, it is desirable that it is 60% or more above all 50% or more 40% or more, the rate of welding is this range, and it can be said that the welding nature which was excellent according to the rank is shown.

[0052] Moreover, when the dimensional stability under hot environments etc. is taken into consideration, as for especially the degree of crystallinity, it is desirable that it is about about 20 – 40%. In any [ this ] case, the heat shrink by the temperature change becomes large, and a possibility of generating deformation, camber, etc. produces what degree of crystallinity is less than 20%, or exceeds 40%. Moreover, that to which degree of crystallinity exceeds 40% becomes weak, and also produces the problem that the reinforcement needed as mentioned above is not obtained.

[0053] Various approaches are employable in order to adjust the degree of crystallinity of a foaming object to the predetermined value of above—mentioned within the limits. For example, what is necessary is just to raise degree of crystallinity by holding and heat—treating a foaming object within metal mold for a while, without taking out from metal mold immediately etc., when the degree of crystallinity of the foaming object after foaming is lower than the value made into the purpose.

[0054] Moreover, what is necessary is to quench metal mold and just to control the rise of degree of crystallinity, in being near, the value which the degree of crystallinity of the foaming object just behind foaming makes the purpose, and. The degree of crystallinity of foam is Japanese Industrial Standards JIS as well as the degree of crystallinity of the pre-expansion particle described previously. It asks from the amount of cold heat of crystallization and the amount of heat of fusions which were measured according to the measuring method of K7121 printing.

[0055] After using it for the interior material for automobiles, it decomposes and collects, and the core material as a foaming object can be reused as a pre-expansion particle etc. While being able to contribute to effective reuse-izing of a resource, and streamlining of dust by reusing

a used foaming object in this way, low cost-ization of a foaming object can also be attained.

[0056]

[Example] Hereafter, an example and the example of a comparison are given and the point which is excellent in this invention is explained concretely. In addition, the degree of crystallinity of the foaming object manufactured using a pre-expansion particle and it is Japanese Industrial Standards JIS, as mentioned above. It asked from the result measured according to the measuring method of K7121 printing.

[0057] Moreover, the consistency was measured by the following approach.

Measurement Japanese Industrial Standards JIS of a consistency K6767 was asked for the bulk density (g/cm3) of a pre-expansion particle, and the consistency (g/cm3) of a foaming object by the degree type based on the approach of printing.

[0058]

```
[Equation 2] 密度 (g/cm³) = 発泡体の重量 (g) 

発泡体の意体積 (cm³)
```

[0059] Moreover, about the foaming object as a core material of the interior material for automobiles manufactured in the following examples and the example of a comparison, the following trial was performed and the property was evaluated.

Measurement Japanese Industrial Standards JIS of 5% compressive strength While measuring 5% compressive strength (kg/cm2) in the temperature requirement with an environmental temperature of -20-+50 degrees C of the foaming object of each example and the example of a comparison to A9511 based on the approach of printing, ratios S2/S1 were calculated from the minimum value S1 and maximum S2. [0060] After bending the foaming object of measurement each example of the rate of welding, and the example of a comparison and making it fracture in the thickness direction, counting of the number of all the foaming particles that exist in the fracture surface, and the number of the foaming particle in which the particle itself carried out ingredient destruction was carried out before long. And it asked for the rate of welding used as the criteria of the welding nature of particles (%) by the degree type.

# [0061] [Equation 3] 融着率 (%) = 材料破壊した発泡粒子の個数 ×100

[0062] The polyethylene terephthalate resin pellet 70 weight section which reproduced the example 1 recovery PET bottle, Content rate: 7.3% of the weight of the isophthalic acid, the crystallization peak temperature of 153.9 degrees C, and the thermoplastic polyester system resin [IV value 0.72] 30 weight section containing an isophthalic acid component by which the speed of crystallization was controlled, The pyromellitic acid 2 anhydride 0.3 weight section and the sodium carbonate 0.03 weight section are supplied to an extruder [aperture:65mm, L/D ratio:35]. On conditions with an engine-speed 50rpm [ of a screw ], and a barrel temperature of 270-290 degrees C with melting and mixing a barrel -- on the way -- the butane (n-butane / isobutane = 7/3) as a foaming agent from press fit tubing boiled and connected -- mixture -- receiving -- 1.1 % of the weight — it came out comparatively and pressed fit. [0063] After extruding and carrying out pre-expansion of the mixture of a melting condition next through each nozzle of the multi-nozzle metal mold [that by which 15 nozzles with a diameter of 0.8mm have been arranged on a straight line] connected at the tip of a barrel, it cooled by the cooling water tank immediately held at 20 degrees C. And fully removing the water adhering to the foam of the shape of a cooled strand, the pellet cutter cut and the pre-expansion particle was manufactured. Bulk density was 0.14 g/cm3.

[0064] Next, this pre-expansion particle is put into a proof-pressure well-closed container, a carbon dioxide is introduced, and after pressurizing 0.5MPa(s) (gage pressure) and holding the inside of a container in ordinary temperature for 2 hours, the foaming particle was taken out from the well-closed container, it put in in the foaming tub, and the steam which mixed air was introduced, and the temperature in a foaming tub is kept at 60-70 degrees C, and carried out the recurrence bubble for 120 seconds. the pre-expansion particle obtained here — an approximate circle with a diameter [ of 2.5mm ], and a die length of 2.5mm — it was pillar-shaped, and bulk density was 0.06 g/cm3 and degree of crystallinity was 7.1%. [0065] Next, put this pre-expansion particle into a proof-pressure well-closed container, introduce a compressed air, and the inside of a container

is pressurized at 0.5MPa(s) (gage pressure). After holding in ordinary temperature for 5 hours, the pre-expansion particle taken out from the well-closed container immediately It has the configuration shown in drawing 1. And 500mm of \*\*\*\*, horizontal abbreviation of 800mm, thickness of about 10mm of the subject section, The metal mold for manufacturing the foaming object as a core material 1 of the door trim for automobiles with a maximum thickness of about 30mm is filled up. A mold clamp meal, Welding was carried out at the same time it subsequently introduced the steam of gage pressure 0.08MPa for the steam of gage pressure 0.04MPa for 15 seconds for 10 seconds and carried out heating expansion of the pre-expansion particle into this mold.

[0066] When the skin temperature of the metal mold which touches the foaming object immediately after steam installation termination was measured, it was 117 degrees C. And the foaming object which was held (the skin temperature of metal mold falls to 104 degrees C) and which carries out after water cooling and has said dimension and a configuration was manufactured for 100 seconds in this condition. The consistency of the acquired foaming object was 0.06g/ml, and degree of crystallinity was 29.2% in the core 27.9% in the epidermis section. Moreover, the rate of welding showed 81% and good welding nature.

[0067] Moreover, the 5% compressive strength of a foaming object was as follows, the ratios S2/S1 calculated from the minimum value S1 and maximum S2 are 1.25, and having related almost fixed impact absorptivity ability to temperature was checked.

(5% compressive strength)

- With polypropylene foam 20 times the example of 20 degree-C:2.5 kg/cm20 degree-C:2.3kg/cm223 degree-C:2.1 kg/cm250 degree-C:2.0 kg/cm2 comparison 1 expansion ratio of this, the shape of an example 1 and isomorphism and the foaming object of this dimension were manufactured. And when compressive strength was measured the 5%, it was as follows, and the ratios S2/S1 calculated from the minimum value S1 and maximum S2 are 3.11, and it became clear that impact absorptivity ability was sharply changed according to a temperature change. (5% compressive strength)
- The result beyond 20 degree-C:5.6 kg/cm20 degree-C:3.7 kg/cm223 degree-C:2.7 kg/cm250 degree-C:1.8 kg/cm2 is shown in Table 1. [0068]

# [Table 1]

		実施例1	比較例1	
	-20°C	2.5	5.6	
5%圧縮強度	30	2,3	3.7	
(kg/cm²)	23°C	2.1	2.7	
(Kg/CIII/	50°C	50°C 2.0 S <sub>2</sub> /S <sub>1</sub> 1.25	1.8	
	$S_2/S_1$		3.11	

[Effect of the Invention] As mentioned above, as explained in full detail, according to this invention, the characteristic effectiveness that the interior material for automobiles which has related almost fixed impact absorptivity ability to temperature can be offered is done so.

[Translation done.]

#### (19) 日本国特許庁 (JP)

# (12) 公開特許公報(A)

(11)特許出願公開番号 特開2001-26246 (P2001 - 26246A)

(43)公開日 平成13年1月30日(2001.1.30)

(51) Int.Cl. <sup>7</sup>		識別記号	FΙ	5	テーマコード(参考)
B60R	21/04		B 6 0 R 21/04	В	3 D 0 2 3
B 3 2 B	5/18		B 3 2 B 5/18		3 J 0 6 6
C08J	9/00		C08J 9/00	Z	4F074
F 1 6 F	7/00		F16F 7/00	· <b>J</b>	4F100
# B29C	44/00		B 6 0 R 13/02	Z	4 F 2 1 2
_		<b>審査</b> 記	請求 未請求 請求項の数4	OL (全 9 頁)	最終頁に続く
(21)出願番号	}	<b>特願平11-198021</b>	(71)出願人 00000	2440	
(22)出願日	•	平成11年7月12日(1999.7.12)	大阪市	積水化成品工業株式会社 大阪市北区西天満二丁目4番4号 (72)発明券 江口 海地	

栃木県下都賀郡野木町潤島800-220

(72)発明者 小野 靖裕

茨城県猿島郡総和町下辺見1266

(74)代理人 100075155

弁理士 亀井 弘勝 (外2名)

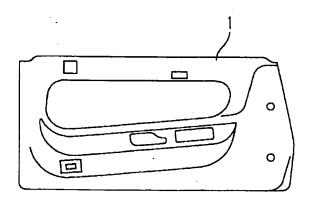
最終頁に続く

#### (54)【発明の名称】 自動車用内装材

#### (57)【要約】

【課題】 温度に関係なくほぼ一定の衝撃吸収性能を有 する自動車用内装材を提供する。

【解決手段】 自動車用内装材の芯材1を、環境温度-20~+50℃の温度範囲での、5%圧縮強度の最小値 S1と最大値S2との比S2/S1が3以下である合成樹脂 の発泡体にて形成した。



#### 【特許請求の範囲】

【請求項1】合成樹脂の発泡体からなり、環境温度-2 0~+50℃の温度範囲での、5%圧縮強度の最小値S 1と最大値S2との比S2/S1が3以下である芯材を備えることを特徴とする自動車用内装材。

【請求項2】芯材が、熱可塑性ポリエステル系樹脂の予備発泡粒子を型内発泡成形した発泡成形体にて形成されており、その結晶化度が20~40%、融着率が40%以上である請求項1記載の自動車用内装材。

【請求項3】熱可塑性ポリエステル系樹脂が、その全成分中に、イソフタル酸、およびシクロヘキサンジメタノールからなる群より選ばれた少なくとも1種の成分を、総量で0.5~10重量%の範囲で含有するものである請求項2記載の自動車用内装材。

【請求項4】熱可塑性ポリエステル系樹脂の少なくとも一部が、使用済みの製品から回収した再生樹脂である請求項2または3記載の自動車用内装材。

#### 【発明の詳細な説明】

[0001]

【発明の属する技術分野】本発明は、自動車用内装材に 関するものである。

[0002]

【従来の技術】たとえばドア内面、ドアトリム、天井下面、リアパッケージ、ニーボルスター、エアバッグドア、ヘッドレスト、アームレスト、各種ピラー、クォータートリム、フロントサイドトリム、フロントシートパック、クラッシュパッド、コンソールボックス、コンソールボックス・コンソールボックス・コンソールボックスをどの自動車用内装材として近時、ポリプロピレン系樹脂製の型内発泡成形体からなる芯材と、当該芯材ををといる高発泡倍率のポリオレフィン系樹脂発泡シート製の表皮材とを備えたものが提案された(たとえば特開平6-115025号公報参照)。

#### [0003]

【発明が解決しようとする課題】ところが、発明者らが 検討したところによると上記の自動車用内装材において 芯材を構成するポリプロピレン系樹脂の発泡体は、軽量 性、耐割れ性、断熱性、耐薬品性などにすぐれるもの の、機械的強度、とくに圧縮強度の温度依存性が大きい ために、たとえば自動車の衝突時などに、中に乗ってい る運転者や添乗者を保護すべく、その衝撃を吸収する性 能の温度依存性が高く、とくに高温環境下での衝撃吸収 性能が不足すること、それゆえ温度上昇による強度低下 を考慮したスペック設計、具体的にはより密度の大きな 発泡体を使用するなどの必要が生じて、十分な軽量化が 図れないといった種々の問題を生じることが明らかとなった。

【0004】本発明の目的は、温度に関係なくほぼ一定

の衝撃吸収性能を有する自動車用内装材を提供すること にある。

[0005]

【課題を解決するための手段】上記課題を解決するために、発明者らは自動車用内装材の芯材がどのような特性を有していれば、温度に関係なくほぼ一定の衝撃吸収性能を有する自動車用内装材が得られるかについて検討を行った。その結果、自動車を、年間を通して通常に使用した際に内装材が経験するであろうと予測されるおよそー20~+50℃程度の温度範囲での、5%圧縮強度の最小値S1と最大値S2との比S2/S1が3以下であれば、そのような芯材を用いた自動車用内装材は、衝突時などに衝撃を吸収する性能を、温度に関係なくほぼ一定に維持できることを見出し、本発明を完成するに至った。

【0006】すなわち本発明の自動車用内装材は、合成 樹脂の発泡体からなり、環境温度-20~+50℃の温 度範囲での、5%圧縮強度の最小値 $S_1$ と最大値 $S_2$ との 比 $S_2/S_1$ が3以下である芯材を備えることを特徴とす るものである。

[0007]

【発明の実施の形態】以下に、本発明を説明する。

〈自動車用内装材〉本発明の自動車用内装材は、前述したように合成樹脂の発泡体からなる芯材を備えており、当該芯材の、環境温度-20~+50℃の温度範囲での、5%圧縮強度の最小値S1と最大値S2との比S2/S1が3以下であることを特徴とするものである。

【0008】上記比S2/S1が3を超えるものは、前述したように、たとえば衝突時の衝撃吸収性能が環境温度によって大きく変動するなどの問題を生じる。なお自動車用内装材の衝撃吸収性能を、環境温度の変化に対してより一層、安定させるためには、上記比S2/S1が2以下であるのが好ましく、1.5以下であるのがさらに好ましい。

【0009】なお比S2/S1の下限値は、いうまでもなく、上記最小値S1と最大値S2とが等しい場合の1である。かかる温度一圧縮強度特性を満足する合成樹脂の発泡体としては種々考えられるが、とくに熱可塑性ポリエステル系樹脂の予備発泡粒子を型内発泡成形した発泡成形体が好適に使用される。

【0010】熱可塑性ポリエステル系樹脂の発泡成形体は上記の温度一圧縮強度特性を満足する上、軽量で、かつ衝撃吸収性、繰り返し圧縮永久歪み、耐薬品性などにすぐれている。また、上記熱可塑性ポリエステル系樹脂の発泡成形体は、とくに高温環境下での寸法安定性にもすぐれており、以下に述べる表皮材、後発泡倍率のシート、形状保持材などの他の部材との寸法、形状のずれなどによる変形やそりなどを発生しにくい、長期の使用に十分に耐えうる自動車用内装材を構成できるという利点を有している。

【0011】自動車用内装材は、上記芯材の、少なくとも自動車の室内に露出する部分を、従来同様に表皮材で被覆するなどして形成される。表皮材としては、前述したポリプロピレン系樹脂シートなどの柔軟なシート材が好適に使用される。また表皮材と芯材との間に、前述した高発泡倍率のポリオレフィン系樹脂発泡シートなどを介装すると、芯材による衝撃吸収性能をそのまま維持しつつ、内装材を押圧したときの感触などを向上できるという利点がある。

【0012】また自動車用内装材には、その全体の形状を維持するために、板状あるいは枠状などに形成された形状保持材を組み込んでもよい。その他、本発明の要旨を変更しない範囲で、種々の設計変更を施すことができる。

〈熱可塑性ポリエステル系樹脂〉本発明において芯材に 好適に使用される発泡成形体を形成する、前記の熱可塑 性ポリエステル系樹脂としては、たとえばテレフタル酸 とエチレングリコールとを重縮合反応させるなどして合 成されるポリエチレンテレフタレート(PET)に代表 される、従来公知の種々の熱可塑性ポリエステル系樹脂 が、いずれも使用可能である。

【0013】ただし、上記PETなどの従来の熱可塑性ポリエステル系樹脂は一般にガスバリヤ一性が高く、発泡剤を含浸するのに多大な時間を要するために、樹脂に発泡剤を含浸させ〔含浸工程〕、ついで加熱して予備発泡させるとともに、粒子化して予備発泡粒子を得たのち〔予備発泡工程〕、この予備発泡粒子を型内に充てんし、加熱膨張させて発泡成形〔型内発泡工程〕する従来の発泡成形方法によって発泡成形体、すなわちこの場合は自動車用内装材の芯材を製造したのでは時間、コストおよび手間がかかるおそれがある。

【0014】さらに上記従来の熱可塑性ポリエステル系樹脂は加熱によって結晶化が進みやすい、すなわち結晶化の速度が速いために、上記含浸時や予備発泡時の加熱によって予備発泡粒子の結晶化度が過度に高くなって、型内発泡成形時に発泡粒同士の発泡融着性の低下をまねくという問題もある。このため、とくに汎用の発泡成形機を使用して、たとえばスチームのゲージ圧が0.5MPa以下といった通常の成形条件で発泡成形したのでは、耐熱性にすぐれた発泡成形体が得られるものの、発泡粒同士が高い融着率でもって良好に融着、一体化した、前記圧縮強度などの強度にすぐれた発泡成形体を製造することはできない。

(

【0015】したがってPETなどの従来の熱可塑性ポリエステル系樹脂を用いて、高い融着率を有する発泡成形体を製造するには、たとえば多量のスチームを型内に均一に供給できるなどの特殊な機能を付与した特殊な発泡成形機を用いて、ゲージ圧が0.5MPaを超えるような特殊な成形条件で成形を行う必要を生じる。しかしながらこのような特殊な成形条件ゆえに、製造される発

泡成形体は、たとえば40%を超えるような、過度に結晶化度の高いものとなってしまい、耐熱性にはすぐれるものの脆くなって、かえって必要とする強度が得られない。

【0016】また、結晶化度が40%を超えた発泡成形体は、とくに高温環境下での寸法安定性が低下して、内装材の変形やそりなどを生じやすいという問題もある。それゆえ本発明においては、上記熱可塑性ポリエステル系樹脂として、とくにその結晶化の速度が抑制されたものを使用するのが好ましい。すなわち結晶化の速度が抑制された熱可塑性ポリエステル系樹脂は、従来のPETなどと比べて、加熱による、予備発泡粒子の結晶化度が過度に高くなることが抑制され、型内発泡成形時の発泡融着性が著しく低くなることが防止される。

【0017】したがって型内発泡成形時の発泡融着性にすぐれ、かつ機械的強度にもすぐれるとともに寸法安定性にもすぐれた発泡成形体を、特殊な発泡成形機を使用することなく、汎用の発泡成形機を使用した通常の成形条件によって、容易に製造することが可能となり、また得られた発泡成形体の結晶化度が過度に高くなることを抑制することができる。

【0018】熱可塑性ポリエステル系樹脂の結晶化の速度は、示差走査熱量計(DSC)を使用して、日本工業規格JIS K7121所載の測定方法に準じて測定した樹脂の結晶化のピーク温度(昇温時に結晶化が起こるピークの温度)によって評価することができる。すなわち結晶化のピーク温度が高いほど樹脂は、結晶化を促進させるのに多量の熱を必要とする、つまり結晶化の速度が遅いと言える。

【0019】具体的には、測定試料としての所定量の熱可塑性ポリエステル系樹脂をDSCの測定容器に充てんして、10℃/分の昇温速度で昇温しながら、上記結晶化ピーク温度が測定される。このようにして測定された熱可塑性ポリエステル系樹脂の結晶化ピーク温度の範囲がおよそ130℃以上であれば、前記のように結晶化の速度が抑制された、好適な熱可塑性ポリエステル系樹脂であるといえる。

【0020】なお結晶化ピーク温度は、上記の範囲内でもとくに180℃以下であるのが好ましい。結晶化ピーク温度が180℃を超えた場合には、樹脂のガラス転移点が高くなるために型内発泡成形の条件幅が狭くなって却って成形が容易でなくなる上、型内発泡成形時に、成形体の表面に収縮が発生しやすくなって外観の良好な発泡成形体が得られないという問題を生じるおそれもある。また、製造された発泡成形体が脆くなってしまうという問題も生じうる。

【0021】なお上記各特性のバランスを考慮して、良好な予備発泡粒子、ならびに良好な発泡成形体を製造することを考慮すると、熱可塑性ポリエステル系樹脂のピーク温度は、上記の範囲内でもとくに132~175℃

程度であるのが好ましく、135~170℃程度であるのがさらに好ましい。かかる特性を満足する熱可塑性ポリエステル系樹脂としては、これに限定されないがたとえば、その全成分中に、イソフタル酸、およびシクロヘキサンジメタノールからなる群より選ばれた少なくとも1種の成分を、総量で0.5~10重量%の範囲で含有するものがあげられる。

【0022】すなわち、ジカルボン酸として、式(1): 【0023】

【0024】で表されるイソフタル酸を使用するか、あるいはジオールとしてシクロヘキサンジメタノールを使用するか、またはこの両者を併用するとともに、いずれか一方を単独で使用する場合はその単独での含有割合を、また両者を併用する場合はその合計の含有割合を、また両者を併用する場合はその合計の含有割合を、それぞれ全成分中の、0.5~10重量%の範囲内とした上記の熱可塑性ポリエステル系樹脂は、イソフタルをおよび/またはシクロヘキサンジメタノールの持つ、樹脂の結晶化を抑制する作用によって、結晶化ピーク温度が130~180℃の範囲内となるため、前記のようとが可能となる。

【0025】なおイソフタル酸および/またはシクロヘキサンジメタノールの含有割合は、上記各特性のパランスを考慮して、良好な予備発泡粒子、ならびに良好な発泡成形体を製造することを考慮すると、上記の範囲内でもとくに0.6~9.5重量%程度であるのが好ましく、0.7~9重量%程度であるのがさらに好ましい。上記のうちシクロヘキサンジメタノールとしては、基本的に、2つのメタノール部分がそれぞれシクロヘキサン環の1位と4位に置換した、式(2):

[0026]

【化2】

【0027】で表される1,4ーシクロヘキサンジメタノールが使用されるが、2つのメタノール部分がシクロヘキサン環の他の位置に置換した異性体も、少量であれば併用可能である。上記イソフタル酸、およびシクロヘキサンジメタノールとともに熱可塑性ポリエステル系樹脂を構成する他の成分のうちジカルボン酸としては、たとえばテレフタル酸やフタル酸などがあげられる。

【0028】またジオール成分としては、たとえばエチレングリコール、 $\alpha$ ーブチレングリコール(1, 2ーブタンジオール)、 $\beta$ ーブチレングリコール(1, 3ーブタンジオール)、テトラメチレングリコール(1, 4ーブタンジオール)、2, 3ーブチレングリコール(2,

3 - ブタンジオール)、ネオペンチルグリコールなどが あげられる。

【0029】また熱可塑性ポリエステル系樹脂の原料には、上記の各成分に加えて、たとえば酸成分として、トリメリット酸などのトリカルボン酸、ピロメリット酸などのテトラカルボン酸などの、三価以上の多価カルボン酸やその無水物、あるいはアルコール成分として、グリセリンなどのトリオール、ペンタエリスリトールなどセリンなどのトリオール、ペンタエリスリトールなどでトラオールなどの、三価以上の多価アルコールなどを、前述した、熱可塑性ポリエステル系樹脂の結晶性や結晶化の速度などに影響を及ぼさない範囲で少量、含有させてもよい。

【0030】上記の熱可塑性ポリエステル系樹脂は、上記の各成分を所定の割合、つまり前記のようにイソフタル酸および/またはシクロヘキサンジメタノールを、総量で0.5~10重量%の範囲で含有した原料を、従来同様に重縮合反応させることによって製造される。また上記熱可塑性ポリエステル系樹脂は、イソフタル酸および/またはシクロヘキサンジメタノールの含有割合の全域分中に占めるイソフタル酸および/またはシクロヘキサンジメタノールの含有割合が、総量で0.5~10重量%の範囲内となるように配合し、たとえば押出機などを用いて、加熱下で溶融、混合することによっても製造できる。

【0031】この方法によれば、予備発泡粒子の製造段階で、イソフタル酸および/またはシクロヘキサンジメタノールの含有割合の異なる2種以上の熱可塑性ポリエステル系樹脂の配合割合を変更するだけで、製造された予備発泡粒子における上記両成分の含有割合を調整できる。このため、樹脂の合成段階で両成分の含有割合を調整する場合に比べて調整作業を簡略化でき、仕様の変更などに柔軟に対応できるようになるという利点がある。

【0032】また、たとえば配合する熱可塑性ポリエステル系樹脂の1種として、使用済みのペットボトルなどから回収、再生した材料などを使用することにより、資源の有効な再利用化とゴミの減量化、ならびに予備発泡粒子の低コスト化を図ることが可能となるという利点もある。なお上記の方法においては、2種以上の熱可塑性ポリエステル系樹脂間でのエステル交換反応により各樹脂がアロイ化して均一な熱可塑性ポリエステル系樹脂となるように、加熱下で十分に溶融、混合してやるのが好ましい。

【0033】なお予備発泡粒子を、後述するように押出機などを用いて、高圧溶融下、発泡剤と混合したのち予備発泡させ、ついで切断して製造する場合には、上記のように2種以上の樹脂の溶融、混合による均一な熱可塑性ポリエステル系樹脂の作製を、発泡剤の混合に先だって上記の押出機中で行い、ついで連続して、上記の製造方法を実施するのが、効率的であり好ましい。

【0034】ただし、あらかじめ別の装置を用いて2種以上の樹脂を溶融、混合して作製しておいた均一な熱可塑性ポリエステル系樹脂を押出機に投入して、上記の製造方法により予備発泡粒子を製造しても構わない。なお本発明で使用する熱可塑性ポリエステル系樹脂は、予備発泡粒子を製造する際の溶融、混合性や、製造された予備発泡粒子を製造する際の溶融、混合性や、製造された予備発泡粒子を用いて、型内発泡成形によって発泡成形体を製造する際の成形性などを考慮すると、その固有粘度(測定温度:35℃、溶媒:オルソクロロフェノール)が0.6~1.5程度であるのが好ましい。

〈予備発泡粒子〉予備発泡粒子は、従来同様に、上記の 熱可塑性ポリエステル系樹脂に発泡剤を含浸させたの ち、加熱して予備発泡させるとともに粒子化して製造し てもよい。

【0035】ただし、熱可塑性ポリエステル系樹脂に発泡剤を含浸させる工程を省略して時間、コストおよび手間を省くとともに、製造される予備発泡粒子の結晶化度をさらに低くして、型内発泡成形時の発泡融着性の低下をさらに抑制するためには、前述したように、上記熱可塑性ポリエステル系樹脂を高圧溶融下、発泡剤と混合し、予備発泡させて予備発泡体を得たのち、これを切断して予備発泡粒子を製造するのが好ましい。

【0036】熱可塑性ポリエステル系樹脂を高圧溶融下、発泡剤と混合して予備発泡させる方法としては、押出機を用いた押出発泡法が効率的であり、好適に採用される。使用できる押出機はとくに限定されず、通常この種の押出発泡成形に使用される単軸押出機、二軸押出機などであり、さらにはこれらを連結したタンデム型であっても良いが、十分な溶融、混合能力を有する押出機が好ましい。

【0037】押出機の口金としてはいろいろなものを使用することができる。たとえば、円環状の口金、フラット口金、ノズル口金、さらには複数のノズルが配置されたマルチノズル口金などがあげられる。これらの口金を使用して、シート状、板状、ロッド状などの、種々の形状の予備発泡体を作ることができる。予備発泡体を、上述した所定の形状とするためには、いろいろな方法が採用される。

【0038】たとえばシート状の予備発泡体を得るには、円環状の口金から押し出された円筒状の予備発泡体を、マンドレル上を進行させてシート状としたり、フラット口金より押し出された厚みのある板状の予備発泡体を、チルロールによりシート状としたりすればよい。ま

た厚みのある板状の予備発泡体を得るためには、一対の 金属板に密接させながら発泡を進行させて、所定の厚み とする方法などが採用される。

【0039】予備発泡体の冷却方法としては、空冷や水冷のほか、温度調整された冷却装置に接触させるなど、いろいろな方法を用いることができる。予備発泡体の冷却はできる限り速やかに行い、結晶化が過度に進行するのを抑制することが重要である。このようにして製造した各種形状の予備発泡体を適宜、切断して円柱状、角状、チップ状などとすることで予備発泡粒子が完成する。

【0040】上記予備発泡体の冷却と切断は、適宜のタイミングで行うことができる。たとえば、口金より押し出された予備発泡体を、発泡中ないし発泡完了後の任意の時点で水中に通すなどして冷却した後、ペレタイザーなどを用いて所定の形状、大きさに切断してもよい。また口金から押し出された、発泡完了直前もしくは発泡完了直後でかつ冷却前の予備発泡体をすぐさま切断したのち、冷却してもよい。

【0041】さらに、シート状に押し出された予備発泡体は、一旦巻き取り機などによってロール状に巻き取って保管した後、粉砕機や切断機にて切断してもよい。かくして製造される予備発泡粒子の大きさは、平均粒径で表しておよそ0.5~5mm程度が好ましい。また予備発泡粒子の結晶化度は、前記のように汎用の発泡成形機を使用して、通常の成形条件で発泡成形した際に、粒子同士の融着性にすぐれた、機械的強度の高い発泡成形体を得ることを考慮するとおよそ8%以下程度であるのが好ましい。

【0042】また、予備発泡粒子をつくる際に、まだ余熱をもっている予備発泡粒子同士が合着しやすくなるのを防止するためには、上記結晶化度は、およそ1%以上であるのが好ましい。なお予備発泡粒子の結晶化度は、上記の範囲内でもとくに1~7%程度であるのが好ましく、1~6%程度であるのがさらに好ましい。

【0043】結晶化度(%)は、先に述べた結晶化ピーク温度の測定と同様に、示差走査熱量計(DSC)を使用して、日本工業規格JIS K7121所載の測定方法に準じて測定した冷結晶化熱量と融解熱量とから、次式によって求められる。

【0044】 【数1】

結晶化度(%) = モルあたりの融解系量ーモルあたりの冷結晶化熱量 完全結晶PETのモルあたりの融解熱量

【0045】なお式中の、完全結晶PETのモルあたりの融解熱量は、高分子データハンドブック [培風館発行]の記載から26.9kJとする。具体的には、測定試料としての所定量の予備発泡粒子をDSCの測定容器に充てんして、10℃/分の昇温速度で昇温しながら冷

結晶化熱量と融解熱量とを測定し、その測定結果から、 上記式に基づいて予備発泡粒子の結晶化度が求められる。

【 0 0 4 6 】 予備発泡粒子の嵩密度は、当該予備発泡粒子を型内発泡成形して製造される発泡成形体の密度など

に応じて適宜、調整できるが、通常は、発泡成形体とほぼ等しい嵩密度であるのが好ましい。予備発泡粒子には、いろいろな添加剤を添加してもよい。添加剤としては、発泡剤の他に、たとえば気泡調整剤、難燃剤、帯電防止剤、着色剤などがあげられる。また、熱可塑性ポリエステル系樹脂の溶融特性を改良するために、グリシト酸ニ無水物のような酸無水物、炭酸ナトリウムのようなである。とくは二種以上混合して添加することができる。とくにこれらの改質剤は、予備発泡粒子の独立気泡率を向上するため、予備発泡粒子の膨張力を大きくできるので有効である。

【0047】本発明で使用できる発泡剤としては、大別 すると、熱可塑性ポリエステル系樹脂の軟化点以上の温 度で分解してガスを発生する固体化合物、加熱すると熱 可塑性ポリエステル系樹脂内で気化する液体、加圧下で 熱可塑性ポリエステル系樹脂に溶解させ得る不活性な気 体などに分類されるが、このいずれを用いてもよい。こ のうち固体化合物としては、たとえばアゾジカルポンア ミド、ジニトロソペンタメチレンテトラミン、ヒドラゾ ルジカルボンアミド、重炭酸ナトリウムなどがあげられ る。また気化する液体としては、たとえばプロパン、n ーブタン、イソブタン、n-ペンタン、イソペンタン、 ヘキサンのような飽和脂肪族炭化水素、ベンゼン、キシ レン、トルエンのような芳香族炭化水素、塩化メチル、 フレオン(登録商標)のようなハロゲン化炭化水素、ジ メチルエーテル、メチルーtert – ブチルエーテルの ようなエーテル化合物などがあげられる。さらに不活性 な気体としては、たとえば二酸化炭素、窒素などがあげ られる。

【0048】なお予備発泡粒子を、前述したように押出機を用いて高圧溶融下、発泡剤と混合し、押し出して予備発泡させたのち、切断して熱可塑性ポリエステル系樹脂予備発泡粒子を製造する場合には、押出機の口金から押し出された瞬間に気化して溶融樹脂を発泡させるとともに、当該溶融樹脂の熱を奪う発泡剤、たとえば飽和脂肪族炭化水素、ハロゲン化炭化水素などを使用するのが好ましい。これらの発泡剤は、溶融した熱可塑性ポリエステル系樹脂を冷却する作用をし、予備発泡粒子の結晶化度を低く抑える効果があるため好ましい。

【0049】また予備発泡粒子には、熱可塑性ポリエステル系樹脂の結晶性や結晶化の速度に大きな影響を及ぼさない範囲で、たとえばポリプロピレン系樹脂などのポリオレフィン系樹脂、ポリエステル系などの熱可塑性エラストマー、ポリカーボネート、アイオノマーなどを添加してもよい。予備発泡粒子を用いて、発泡成形体としての自動車用内装材の芯材を製造する方法としては、閉鎖しうるが密閉し得ない金型に予備発泡粒子を充てん

し、さらに加熱媒体としてスチームを導入して型内発泡 成形する方法が好ましい。

【0050】このときの加熱媒体としては、スチーム以外にも熱風やオイルなどを使用することができるが、効率的に成形を行う上ではスチームが最も有効である。成形した発泡成形体は、冷却したのち金型から取り出せばよい。スチームで型内発泡成形する場合には、前述したように汎用の発泡成形機を使用して、通常の成形条件で発泡成形すればよい。すなわち予備発泡粒子を金型へ充てんした後、まず低圧〔たとえばゲージ圧0.04MPa程度〕で一定時間、スチームを金型内へ吹き込んで、粒子間のエアーを外部へ排出する。ついで、吹き込むスチームの圧を昇圧〔たとえば0.08MPa程度〕して、予備発泡粒子を型内発泡させるとともに粒子同士を融着せしめて発泡成形体とすることができる。

【0051】また予備発泡粒子を、あらかじめ密閉容器に入れて、炭酸ガス、窒素、ヘリウム等の不活性ガスを圧入した後、金型での型内発泡成形に使用する直前まで、圧入したガスの雰囲気下に保持することで、予備発泡粒子の、金型での型内発泡成形時の膨張力をより大きくして、良好な発泡成形体を得ることもできる。かくして得られた発泡成形体における、粒子同士の融着性の基準となる融着率は40%以上、とくに50%以上、なかんずく60%以上であるのが好ましく、融着率がこの範囲で、格別に優れた融着性を示すといえる。

【0052】またその結晶化度は、とくに高温環境下での寸法安定性などを考慮すると、およそ20~40%程度であるのが好ましい。結晶化度が20%未満であるか、または40%を超えるものは、このいずれの場合にも、温度変化による熱収縮が大きくなって、変形やそりなどを発生するおそれが生じる。また結晶化度が40%を超えるものは脆くなって、前記のように必要とする強度が得られないという問題も生じる。

【0053】発泡成形体の結晶化度を上記範囲内の所定の値に調整するには、種々の方法を採用することができる。たとえば、発泡成形後の発泡成形体の結晶化度が目的とする値よりも低い場合には、発泡成形体を金型からすぐに取り出さずにしばらくの間、金型内で保持して熱処理することなどによって結晶化度を上昇させてやればよい。

【0054】また、発泡成形直後の発泡成形体の結晶化度が目的とする値と近い場合には、金型を急冷するなどして結晶化度の上昇を抑制してやればよい。発泡体の結晶化度は、先に述べた予備発泡粒子の結晶化度と同じく、日本工業規格JIS K7121所載の測定方法に準じて測定した冷結晶化熱量と融解熱量とから求められる

【0055】発泡成形体としての芯材は、自動車用内装材に使用したのち分解、回収して、予備発泡粒子などとして再利用することが可能である。使用済みの発泡成形

体をこのように再利用することにより、資源の有効な再利用化とゴミの減量化に貢献できるとともに、発泡成形体の低コスト化を図ることもできる。

[0056]

【実施例】以下、実施例、比較例をあげて、この発明のすぐれている点を具体的に説明する。なお、予備発泡粒子とそれを用いて製造した発泡成形体の結晶化度は、前述したように日本工業規格JIS K7121所載の測定方法に準じて測定した結果より求めた。

【0057】また密度は、下記の方法で測定した。 密度の測定

日本工業規格 JIS K6767に所載の方法に準拠して、次式により、予備発泡粒子の嵩密度(g/cm³)、および発泡成形体の密度(g/cm³)を求めた。

【0058】 【数2】

> 密度 (g/cm³) = <u>発泡体の</u>
> 量量 (g) **発泡体の** 高体積 (cm³)

【0059】また、以下の実施例、比較例で製造した自動車用内装材の芯材としての発泡成形体について、下記の試験を行って、その特性を評価した。

5%圧縮強度の測定

日本工業規格 J I S A 9 5 1 1 に所載の方法に準拠して、各実施例、比較例の発泡成形体の、環境温度 - 2 0 ~+50℃の温度範囲での、5%圧縮強度(kg/cm2)を測定するとともに、その最小値 S1と最大値 S2とから、比 S2/S1を求めた。

【0060】融着率の測定

各実施例、比較例の発泡成形体を折り曲げて厚み方向に破断させたのち、破断面に存在する全ての発泡粒子の個数と、そのうち粒子自体が材料破壊した発泡粒子の個数とを計数した。そして次式により、粒子同士の融着性の基準となる融着率(%)を求めた。

【0061】 【数3】

#### 【0062】実施例1

回収ペットボトルを再生したポリエチレンテレフタレート樹脂ペレット70重量部と、イソフタル酸成分を含有する、結晶化のスピードが抑制された熱可塑性ポリエステル系樹脂〔イソフタル酸の含有割合:7.3重量%、結晶化ピーク温度153.9℃、IV値0.72〕30重量部と、ピロメリット酸二無水物0.3重量部と、炭ソーダ0.03重量部とを押出機〔口径:65mm、L/D比:35〕に供給し、スクリューの回転数50rpm、パレル温度270~290℃の条件で溶融、混合ながら、パレルの途中に接続した圧入管から、発泡剤としてのブタン(n ーブタン/イソブタン=7/3)を、混合物に対して1.1重量%の割合で圧入した。

【0063】つぎに、溶融状態の混合物を、バレルの先端に接続したマルチノズル金型〔直線上に、直径0.8 mmのノズルが15個、配置されたもの〕の、各ノズルを通して押し出して予備発泡させたのち、直ちに20℃に保持した冷却水槽で冷却した。そして、冷却されたストランド状の発泡体に付着した水を十分に除去しながらペレットカッターによって切断して予備発泡粒子を製造した。嵩密度は0.14g/cm³であった。

【0064】つぎにこの予備発泡粒子を耐圧密閉容器に入れ、二酸化炭素を導入して容器内を0.5 MPa (ゲージ圧)に加圧して、常温で2時間保持したのち、発泡粒子を密閉容器から取り出して発泡槽内に入れ、空気を混合したスチームを導入して、発泡槽内の温度を60~70℃に保って120秒間、再発泡させた。ここで得ら

れた予備発泡粒子は直径2.5mm、長さ2.5mmの 略円柱状であり、嵩密度は0.06g/cm<sup>3</sup>、結晶化 度は7.1%であった。

【0065】つぎにこの予備発泡粒子を耐圧密閉容器に入れ、圧縮空気を導入して容器内を0.5MPa(ゲージ圧)に加圧して、常温で5時間保持したのち、密閉容器から取り出した予備発泡粒子を直ちに、図1に示す形状を有し、かつ縦約500mm、横約800mm、主体部の厚み約10mm、最大厚み約30mmの、自動車用ドアトリムの芯材1としての発泡成形体を製造するための金型に充てんして型締めし、この型内に、ゲージ圧0.04MPaのスチームを10秒間、ついでゲージ圧0.08MPaのスチームを15秒間、導入して予備発泡粒子を加熱膨張させると同時に融着させた。

【0066】スチーム導入終了直後の、発泡成形体に接する金型の表面温度を測定したところ117℃であった。そしてこの状態で100秒間、保持(金型の表面温度は104℃まで低下)したのち水冷して、前記寸法、形状を有する発泡成形体を製造した。得られた発泡成形体の密度は0.06g/m I、結晶化度は表皮部で27.9%、中心部で29.2%であった。また融着率は81%と良好な融着性を示した。

【0067】また、発泡成形体の5%圧縮強度は下記のとおりであり、その最小値 $S_1$ と最大値 $S_2$ とから求めた比 $S_2$ / $S_1$ は1. 25であって、温度に関係なくほぼ一定の衝撃吸収性能を有することが確認された。

(5%圧縮強度)

-20℃: 2. 5 k g/c m<sup>2</sup>

0℃: 2. 3 k g/c m<sup>2</sup>

23℃: 2. 1 kg/cm<sup>2</sup>

50°C: 2. 0 k g / c m<sup>2</sup>

比較例1

発泡倍率 20 倍の発泡ポリプロピレンにて、実施例 1 と同形状、同寸法の発泡成形体を製造した。そしてその 5% 圧縮強度を測定したところ下記のとおりであり、その最小値  $S_1$ と最大値  $S_2$ とから求めた比  $S_2$ / $S_1$ は 3. 11であって、温度変化に応じて衝撃吸収性能が大きく変動することが判明した。

(5%圧縮強度)

-20°C: 5. 6 k g/cm²

0°C: 3. 7 kg/cm²

23°C: 2. 7 kg/cm<sup>2</sup>

50°C: 1. 8 kg/cm<sup>2</sup>

以上の結果を表1に示す。

#### [0068]

【表 1 】

		実施例1	比較例1
	_20℃	2.5	5.6
5%圧縮強度	20	2.3	3.7
(kg/cm²)	23°C	2.1	2.7
(NB/CIII /	50℃	2.0	1.8
	S <sub>2</sub> /S <sub>1</sub>	1.25	3.11

【発明の効果】以上、詳述したように本発明によれば、 温度に関係なくほぼ一定の衝撃吸収性能を有する自動車 用内装材を提供できるという特有の効果を奏する。

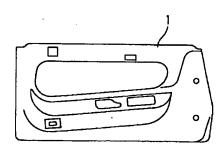
#### 【図面の簡単な説明】

【図1】本発明の実施例、比較例で製造した自動車用ドアトリムの芯材としての発泡成形体の、外観を示す正面図である。

【符号の説明】

1 芯材

【図1】



#### フロントページの続き

(51) Int. CI. 7

識別記号

FΙ

テーマコード(参考)

B29C 67/22

B60R 13/02 B29K 67:00 Fターム(参考) 3D023 BA01 BA07 BB03 BB08 BB09 BC01 BD01 BD03 BD08 BD32

BD33 BE04 BE10 BE31

3J066 AA01 AA23 BA04 BB01 BC01

BD05

4F074 AA65 AA66 BA37 BA38 CA22

CA23 CA32 CA34 CA38 CA49

CC03Y CC03Z CC04X CC04Y

CC04Z CC05Z CC22X CC32Y

CC32Z CC34Z CC47 DA02

DA08 DA24 DA35

4F100 AH01 AK01A AK41A AK42

BA01 CA01 DJ01A EJ02

EJ39 GB33 JB16A JK05A

JK10 JL16 JL16A YY00A

4F212 AA24 AA50 AB02 AE07 AG01

AG20 AH26 UA01 UB01 UF01